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IS : 12502 -1988

( Reaffirmed 2004 )

# Indian Standard

# SPECIFICATION FOR GLYPHOSATE, TECHNICAL

( First Reprint DECEMBER 1996 )

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**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARC
NEW DELHI 110002

Gr 2

May 1989

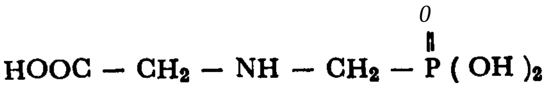
# *Indian Standard*

# SPECIFICATION FOR GLYPHOSATE, TECHNICAL

## 0. FOREWORD

**0.1 This** Indian Standard was adopted by the Bureau of Indian Standards on 24 October 1988, after the draft finalized by the Pest Control Sectional Committee had **been approved by** the **Agricultural and Food Products** Division Council.

0.2 Glyphosate is the accepted common name by International Organization for Standardization (ISO) for *N*-phosphonomethyl glycine. The empirical and structural formulae and molecular mass of glyphosate are indicated below:

| *Empirical Formula* | *Structural Formula* | *Molecular Mass* |
|---|---|---|
| $C_3H_8NO_5P$ |  $HOOC-CH_2-NH-CH_2-\overset{\overset{O}{\parallel}}{P}(OH)_2$ | 169'1 |

**0.3 Glyphosate, technical is used** in the preparation of weedicidal preparations.

**0.4** In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, 'expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values ( *revised* ).

## 1. SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for glyphosate, technical.

## 2. REQUIREMENTS

**2.1 Description** — The material shall essentially consist of N-phosphonomethyl glycine and. shall be in the form of white to off-white powder.

2.2 The material shall also comply with the requirements given in Table 1.

## 3. PACKING AND MARKING

3.1 Packing — Material shall be packed according to the requirements given in IS : 8190 ( Part 1 )-1980*.

**3.2 Marking** — The container shall bear legibly and indelibly the following information in addition to any other information as required

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*Requirements for packing of pesticides: Part 1 Solid pesticides ( *first revision* ).

TABLE 1 REQUIREMENTS FOR GLYPHOSATE, TECHNICAL

( *Clauses* 2.2 *and* 5.1 )

| Sl *No.* | Characteristic | Requirement | Method of Test, Ref to | |
|---|---|---|---|---|
| | | | Appendix of This Standard | Clause No. of IS : 6940-1982* |
| (1) | (2) | (3) | (4) | (5) |
| i) | Glyphosate content, percent by mass, Min | 95·0 | A | — |
| ii) | Extra acidity, percent by mass, *Max* ( *see* Note ) | 1·0 | A-2 | |
| iii) | Chloride ( as NaCl ), percent by mass, *Max* | 1·5 | B | — |
| iv) | Moisture content, percent by mass, Max | 1·0 | — | 4 |

Note — Extra acidity = Total acidity — glyphosate acidity.

*Methods of test for pesticides and their formulations ( *first revision* ).

under *Insecticides Act,* 1968 and Rules framed thereunder:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net mass of contents;
f) Glyphosate content, percent ( *m/m* ); and
g) The minimum cautionary notice as required under *Insecticides Act, 1968* and Rules framed thereunder.

**3.2.1** The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under as well defined system of inspection, testing and quality control which is devised and supervired by BIS and operated by the producer. Standard marked products are alro continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers *may be* obtained from the Bureau of Indian Standards.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn as prescribed in IS : 10946-1984*.

## 5. TESTS

**5.1** Test shall be carried out in accordance with 2.1, 2.2 and col 4 and 5 of Table 1.

**5.2 Quality of Reagents** — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water ( *see* IS : 1070-1977† ) shall be used where its use as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of sampling for technical grade pesticides.

†Specification for water for general laboratory use ( *second revision* ).

# APPENDIX A

[ *Table* 1, *Item* (i) ]

## DETERMINATION OF GLYPHOSATE CONTENT

## A-0. GENERAL

**A-0.1** For the determination of glyphosate content, two methods, namely, UV-spectrophotometric method and titration method have been prescribed. UV-spectrophotometric method shall be the referee method in case of dispute.

## A-1. UV-SPECTROPHOTOMETRIC METHOD

**A-1.1 Principle** — N-phosphonomethyl glycine is determined by measuring absorbance of the secondary amine-nitrite complex at 243 nm. This method depends on the formation of a nitrosoamine complex with secondary amine under acidic conditions; therefore, any secondary amine may interfere with this method. However, the possibility of any secondary amine being present in glyphosate technical is negligible.

**A-1.2 Apparatus** — UV-visible spectrophotometer.

### A-1.3 Reagents

**A-1.3.1** *Sulphuric Acid* — 1 : 1.

**A-1.3.2** *Sodium Nitrite* — 0·2 N.

**A-1.3.3** *Potassium Bromide* — 25 percent.

**A-1.3.4** *Glyphosate Standard* — of known purity.

### A-1.4 Procedure

**A-1.4.1** *Preparation of Standard Solution*

*A-1.4.1.1* Weigh 0·1 g glyphosate standard to the nearest 0·1 mg into a 100-ml volumetric flask. Add 2 drops of sulphuric acid and dissolve with water by continuous shaking till complete dissolution. Dilute to mark to make the volume.

**A-1.4.1.2** Take with a pipette, 25 ml of aliquot into another 100-ml volumetric flask. Add 50 ml water, 5 ml sulphuric acid, 1 ml potassium bromide, 5 ml sodium nitrite and make up to mark with water, Mix and allow to stand for 30 minutes. Dilute 5 ml of this solution to 100 ml in another volumetric flask.

A-1.4.2 *Preparation of Sample Solution* — Weigh 0·1 g of sample to the nearest 0·1 mg into a 100-ml volumetric flask. Add 2 drops of sulphuric acid and dissolve with water by continuous shaking till complete dissolution. Dilute to mark to make the volume. Then continue as in A-1.4.1.2.

A-1.4.3 *Preparation of Blank Solution* — Take 75 ml of water in a 100-ml volumetric flask. Add 5 ml of' sulphuric acid, 1 ml of potassium bromide, 5 ml of sodium nitrite and make up to mark with water. Mix and allow to stand for

30 minutes. Dilute 5 ml of this solution to 100 ml in another volumetric flask.

A-1.4.4 Measure the absorbance of the sample and standard solutions against reagent blank at 243 nm in 1 cm quartz cells.

### A-1.5 Calculation

$$\text{Glyphosate, percent by mass} = \frac{A_1 \times W}{A \times W_1} \times P$$

where

$A_1$ = absorbance for sample solution,
$A$ = absorbance for standard solution,
$W_1$ = weight of sample,
$W$ = weight for standard, and
$P$ = purity of standard glyphosate.

**NOTE 1 — Primary amines and ammonium salts interfere with this method because they react with sodium nitrite. These interferences may be removed by distillation from a basic solution or by adding sufficient sodium nitrite to complex both the interfering primary amines and the ammonium salts. The primary amine-nitrite complex decomposes to nitrogen. Any secondary amine capable of forming a nitrosoamine can interfere.**

**NOTE 2 — The quantity of sodium nitrite to be added as given in the procedure ensures that the reaction between the secondary amine and the nitrite proceeds rapidly.**

**NOTE 3 — The amount of potassium bromide added to the solution does not seem to be significant as long as constant amount is added to each solution before or during the addition of sodium nitrite.**

**NOTE 4 — Acidic conditions are necessary for complete reaction with the nitrite.**

## A-2. TITRATION METHOD

A-2.0 This method is used for total acidity, glyphosate content and extra acidity.

A-2.1 Principle — Total acidity is first estimated using standard alkali by back titration. Acidity due to glyphosate is then estimated on the basis that glyphosate reacts with carbon disulphide to give dithiocarbamix acid derivative of glyphosate which is titrated against standard alkali. Extra acidity is calculated by subtracting acidity due to glyphosate from total acidity.

A-2.2 **Apparatus** — Magnetic stirrer.

A-2.3 Reagents

A-2.3.1 ***Sodium Hydroxide*** — 1 N.

A-2.3.2 ***Hydrochloric Acid*** — 1 N.

**A-2.3.3** ***Phenolphthalein Indicator*** — 1 percent in pyridine.

A-2.3.4 ***Pyridine*** — reagent grade.

A-2.3.5 ***Carbon Disulphide*** — reagent grade.

A-2.3.6 ***Isopropanol*** — reagent grade.

### A-2.4 Procedure

**A-2.4.1** ***Total Acidity*** — Weigh accurately about 2·0 g of the material in 500-ml conical flask and add 30 ml of 1 N sodium hydroxide. Dissolve and titrate excess of sodium hydroxide against 1 N hydrochloric acid using the phenolphthalein indicator. Carry out a blank determination simultaneously. The sample solution and the blank solution should be retained for further estimation as given in A-2.4.2.

A-2.4.2 To the sample solution in A-2.4.1, add 25 ml each of pyridine and isopropanol. Add 5 ml carbon disulphide, shake and immediately titrate against standard sodium hydroxide. Add sodium hydroxide drop by drop with vigorous stirring using a magentic stirrer and take the end point when the pink colour persists for at least one minute. Similarly follow the same procedure in case of blank as retained in **A-2.4.1.**

### A-2.5 Calculation

a) **Total acidity** as glyphosate, percent by mass ( A ) $= \dfrac{(V_1 - V_2) \times N_1 \times 8.45}{M}$

b) **Glyphosate, percent by mass ( *B* )** $= \dfrac{(V_3 - V_4) \times N_2 \times 8{\cdot}45}{M}$

where

$V_1$ = volume in ml of hydrochloric acid used for blank in (a),
$V_2$ = volume in ml of hydrochloric acid used with sample in (a),
$V_3$ = volume in ml of sodium hydroxide used with sample in (b),
$V_4$ = volume in ml of sodium hydroxide used for blank in (b),
$N_1$ = normality of hydrochloric acid used,
$N_2$ = normality of sodium hydroxide used, and
$M$ = mass in g of the material taken for the test.

c) Extra acidity = Total acidity *(A)* — glyphosate content *(B)*

# APPENDIX B

[ *Table 1, Item* (iii) ]

## DETERMINATION OF CHLORIDE CONTENT

### B-1. REAGENTS

**B-1.1 Silver Nitrate Solution** — 0·1 *N*.

B-1.2 **Potassium** Thiocyanate Solution — 0·1 *N*.

**B-1.3 Ferric Alum Indicator Solution**

**B-1.4 Nitric Acid** — 10 percent ( *v/v* ).

### B-2. PROCEDURE

B-2.1 Weigh accurately about 1’0 g of the sample in a 250-ml conical flask. Add 50 ml water and 10 ml dilute nitric acid. Add 25 ml silver nitrate solution and add 2 ml of the ferric alum indicator solution Titrate against potassium thiocyanate till faint red colour is obtained. Carry out a blank titration also.

### B-3. CALCULATION

**B-3.1** Chloride ( as NaCl ), percent by mass $= \frac{(V_1 - V_2) \times 5{\cdot}846 \times N}{M}$

where

$V_1$ = volume in ml of potassium thiocyanate used for titration of the sample,

$V_2$ = volume in ml of potassium thiocyanate used for titration of blank solution,

$N$ = normality of potassium thiocyanate, and

$M$ = mass in g of the sample taken for the test.

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